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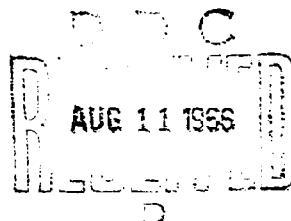
(TITLE UNCLASSIFIED)
DEVELOPMENT OF PROPELLANTS
CONTAINING AN ENERGETIC OXIDIZER

T. P. Rudy
United Technology Center

TECHNICAL REPORT AFRPL-TR-66-154
July 1966

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FOREWORD

(U) This first Quarterly Technical Progress Report under Contract No. AF 04(611)10786 submitted in July 1966, covers work performed during the period 1 June 1965 through 31 May 1966, by United Technology Center, Sunnyvale, California. The contract was initiated under Air Force Rocket Propulsion Laboratory, Research and Technology Division, Project No. 3059. For internal purposes this report is designated UTC 2139-QTR1.

(U) The Air Force program monitor is Karl W. Joffs, 1/Lt., USAF/RPMCP of the Research and Technology Division, Air Force Rocket Propulsion Laboratory, Air Force Systems Command, United States Air Force, Edwards, California 93523.

(U) Principal contributors to the work reported herein are:

J. W. Allan	T. W. Nakagawa
L. S. Bain	G. H. Olsen
R. D. Harline	R. A. Saut
C. J. McCormick	M. E. Steinle

(U) This report contains classified information extracted from the following documents: Final Technical Progress Report, Contract No. AF 04(611)-9894, August 1965, and Quarterly Progress Report No. 2, Contract AF 04(611)-11199, April 1966. Both documents are classified CONFIDENTIAL, downgrading Group 4.

(U) Subject matter contained herein has been placed under Secrecy Order with Permit A by the United States Patent Office. Recipients of this document must take all reasonable safeguards to prevent the unauthorized disclosure of the subject matter. Failure to properly safeguard this information may be punishable by a fine of not more than \$10,000 or imprisonment for not more than two years or both as provided by 35 USC 186.

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CONFIDENTIAL ABSTRACT

(C) United Technology Center (UTC) is conducting a 24-month program to produce the first practical propellants exploiting the high energy and favorable density of nitronium perchlorate (NP). In a previous program under Contract AF 04(611)-9894, a curable binder system was developed and demonstrated using both uncoated and coated particulate NP. Propellants containing uncoated NP had a useful life of only a few days at room temperature, and those using Reta-coated NP were stable for a few months. Original plans for scaled-up motor firings and incorporation of energetic fuel appeared less desirable than emphasis on improvement of stability. During the period of contract redirection only limited effort was undertaken. Because of the unavailability of Reta-coated NP, emphasis has been placed on increasing the useful life of propellants containing uncoated or UTECOATED NP. Means to this end include development of more compatible ingredients and use of reduced temperatures for processing and storage. The latter approach requires methods of decreasing binder viscosity and increasing cure reactivity. Potentially useful hydrocarbon and nitroparaffin plasticizers have been found, and cure catalysis has been effected. Stability studies show that Reta-coated NP has a useful life of less than 1 year under mild storage conditions. Propellants containing UTECOATED NP exhibit unacceptable thermal stability and explosive combustion characteristics after about 1 week at ambient temperature. Formulation studies show that improved ballistic performance is attainable without impairment of stability by incorporation of increased concentrations of aluminum and use of finely divided ammonium perchlorate (AP) as a supplementary oxidizer. Equipment has been constructed for scaled-up drying of NP and for purification of UTREZ prepolymer. Approximately 5 lb of purified prepolymer is on hand. A surveillance facility has been constructed to permit study of propellant stability at reduced temperatures in a dry environment.

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SECTION I

INTRODUCTION

(C) Under Contract No. AF 04(611)-9894 United Technology Center (UTC) conducted a 12-month program to develop a curable binder containing a saturated hydrocarbon, difunctional polymer and to establish the compatibility of this binder with nitronium perchlorate (NP). A further objective of the program was ballistic characterization of the binder/NP formulation in small motors.

(C) These objectives were achieved by the development of a system based on UTREZ prepolymer (α , ω -dicarboxylated polyisobutylene), MRPX plasticizer (a light, isoparaffinic oil), and NTEB crosslinker (a trifunctional aziridinyl compound). Propellants containing both coated and uncoated NP were mixed, cast, and cured at room temperature. Those containing uncoated oxidizer were stable for only a few days at room temperature in a dry environment. Propellants containing Reta-coated oxidizer were found to be stable for periods up to 6 months under the same conditions. General characteristics of the propellants were insensitivity to impact and friction, sensitivity to moisture, limited thermal stability, and reasonable mechanical properties.

(C) All of the propellants were ballistically characterized in micromotor firings, and a formulation containing Reta-coated NP was tested in nominal 1-lb motor firings. Efficient oxidizer utilization, high burning rates, and moderately high pressure exponents were indicated by these tests. The most significant relationships were believed to be those derived from the 1-lb motors:

$$r_b = 0.68 (P_c / 1,000)^{0.49}$$

$$K_n = 320 (P_c / 1,000)^{0.54}$$

(C) Original plans for continued development of NP propellants under the present contract were directed toward increasing the scale of propellant production and motor firings as well as improving ballistic performance by use of higher solids loadings and incorporation of aluminum hydride fuel. However, in light of the marginal stability of the basic propellant system it became necessary to revise these objectives. Formal redirection of the present contract occurred on 3 May 1966. During the 11-month period

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prior to redirection, effort under the contract was maintained at a low level and was limited to activities consistent with both the original contract as well as the anticipated scope of the redirected contract.

(C) A second factor further restricted experimental work during this report period. This was the loss of 6 lb of Reta-coated NP in an explosion during coating operations at Union Carbide Corporation. Because of uncertainty regarding the cause of this incident, Union Carbide took the precaution of destroying 6 remaining pounds of uncoated oxidizer from the lot which was involved in the explosion. The cause of the incident is under intensive study. However, until the cause is understood and means of eliminating this previously unsuspected hazard are in hand, Reta-coated NP will not be available from Union Carbide for use in the present program.

(C) The limited effort which has been conducted during this report period has been directed toward (1) studies of ingredients and formulation techniques intended to improve stability and ballistic performance of NP propellants, (2) development of equipment for purification of ingredients, (3) preparation of supplies of purified ingredients, and (4) construction of surveillance facilities to permit study of propellant stability.

(U) Under the redirected program major emphasis is being placed on improvement of propellant stability, characterization of propellant properties (especially stability), and improvement of ballistic performance.

(C) To achieve the objective of developing practical propellants exploiting the high energy and favorable density of NP requires that the chemical stability and permissible solids loadings of the propellant system be increased. There is little doubt that the useful life of UTREZ/NP propellants can be extended significantly by minimizing thermal exposure. However, use of reduced processing temperature increases viscosity of the binder and thereby decreases attainable solids loadings. Furthermore, the rate of cure of the propellant is decreased as the temperature is lowered. Fortunately, the temperature coefficient of propellant deterioration is greater than that of the crosslinking reaction. The most reasonable approaches to the problem appear to be (1) reduce viscosity of the binder formulation without use of elevated temperature, (2) control the type, characteristics, and amounts of solid ingredients to maximize ballistic performance, (3) reduce the cure temperature to the minimum acceptable value, employing a catalyst if possible, and (4) store the cured propellant at the lowest practical temperature. The ingredient and formulation studies that follow have been conducted to facilitate these approaches.

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SECTION II

INGREDIENT STUDIES

(U) Preliminary studies have been directed toward decreasing the viscosity of the binder system, accelerating the crosslinking reaction, and increasing solids loading.

1. BINDER INGREDIENTS

a. UTREZ Prepolymer

(C) Improvements of the synthesis of UTREZ continue as an important part of UTC's in-house research. Methods of purifying the prepolymer for use with NP received heavy emphasis in the previous contract. In the present program, work dealing with the prepolymer alone has been limited to establishing that material produced in a pilot plant and purified in scaled-up equipment is equivalent in performance to that previously produced in the laboratory and purified on a small scale. The scaled-up purification equipment described in section 6.2 has been employed to purify approximately 5 lb of prepolymer for use in this program.

(C) Future work will include fractionation of the prepolymer to reduce viscosity by removal of high-molecular-weight species. In addition, the use of still more rigorous purification techniques to improve compatibility with NP will be tested.

b. Plasticizers

(C) Of the possible means of decreasing viscosity of UTREZ binder, improved plasticization is particularly attractive. The relatively high viscosity of UTREZ at low temperatures (typically ca 2,500 poise at 25° C) is due only in part to its polyisobutylene backbone. Carboxyl-carboxyl association is believed to be a far more important factor. Evidence supporting this conclusion is the markedly lower viscosity of hydroxy-terminated polyisobutylene and the extreme reduction of viscosity of UTREZ with increasing temperature (ca 13 poise at 95° C). Use of a polar plasticizer would be expected to decrease carboxyl-carboxyl association, and this expectation has been confirmed by experiments with polar compounds of low molecular weight. Unfortunately, most polar,

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but UTREZ-soluble, materials are either incompatible with NP or else they disrupt the crosslinking reaction with NTEB. However, nitroparaffins offer some promise and have the obvious benefit of providing additional oxidizer to the propellant. The latter benefit may be quite important in the achievement of desired O/F ratios.

(U) The effectiveness of several candidate nitroparaffin plasticizers is shown in table I. Although this limited study cannot be regarded as conclusive, it appears that a straight paraffin chain and terminal location of the nitro group is desirable. The non-linear response of viscosity observed with the combination of 1-nitrobutane and MRPX (calculated viscosity, 500 poise; observed, 404 poise) is consistent with the desired interaction of the nitroparaffin with the carboxyl groups of UTREZ. Additional studies with other nitroparaffins are planned.

TABLE I

(U) EFFECT OF NITROPARAFFIN PLASTICIZERS
ON VISCOSITY OF UTREZ*

<u>Plasticizer</u>	Viscosity at 25° C poise
MRPX-1015, 15%w	658
1-Nitrobutane, 15%w	181
MRPX, 10%w/1-Nitrobutane, 5%w	404
2-Nitrobutane, 15%w	267
Nitrocyclohexane, 15%w	520
2, 2-Dinitropropane	Insoluble

* UTREZ not purified

(C) The initial selection of MRPX isoparaffinic plasticizer was based not only on its outstanding compatibility with NP but also on its low volatility (bp above 700° F). In view of the apparent need to avoid exposure of NP propellants to elevated temperatures, the requirement that a plasticizer have a high boiling point is no longer reasonable. Exploratory studies have been conducted to find alternate hydrocarbons with appropriate NP compatibility

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and plasticizing capability greater than that of MRPX. Materials containing branched paraffins and free of both aromatic and olefinic constituents were evaluated (table II). Candidate plasticizers were purified prior to use by the usual sequence of treatments: sulfuric acid extraction, water washing, dissolution in carbon tetrachloride followed by exposure to fuming nitric acid, water washing, drying over anhydrous sodium sulfate then by passage through a column of silica gel, vacuum distillation and, finally, drying over phosphorus pentoxide.

TABLE II
(U) HYDROCARBON PLASTICIZER EVALUATION

<u>Plasticizer (15%w in UTREZ^(a))</u>	<u>Viscosity poise</u>	<u>Compatibility with NP</u>
MRPX-1015 ^(b)	847 at 30° C	Color, 1 week
Humble Marcol 70 ^(c)	660 at 30° C	(Not tested)
Humble Marcol 72 ^(c)	575 at 30° C	Color, 5 min
Shell Spray Base ^(d)	330 at 23.5° C	Color, 2 hr

- (a) Purified
(b) An experimental isoparaffinic oil
(c) A commercial white oil
(d) A hydrotreated petroleum raffinate, bp 185° to 200° C

(C) In addition, two branched hydrocarbon compounds, squalane and 2, 6, 10, 14-tetramethylpentadecane (TMPD) were evaluated. Viscosity determinations using 15%w of the hydrocarbons in unpurified UTREZ gave the following results: MRPX-1015, 658 poise at 25° C; squalane, 945 poise at 23° C; TMPD, 550 poise at 23° C. After purification, squalane initially appeared to be more compatible with NP than MRPX-1015. However, in propellant formulations (discussed in section 4.0) this proved not to be the case. After short contact with NP, TMPD developed noticeable color. However, further degradation appeared to be very slow. When contact with NP was employed as part of the purification procedure, TMPD appeared to be acceptably compatible with NP. On the basis of both compatibility and viscosity, TMPD deserves further study.

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c. Crosslinking Agents - Cure Catalysts

(C) In the preceding program NTEB (nitrilotriethyl- β -ethyleniminobutyrate) proved to be superior to a variety of polyfunctional aziridinyl compounds in terms of both intrinsic compatibility with NP and cure reactivity with UTREZ. However, the study of candidate crosslinkers was far from exhaustive. It is quite possible that even better curatives can be developed.

(C) Earlier studies indicated that tris(methylaziridinyl)triazine (TMAT) is attractive with respect to cure reactivity and NP compatibility. However, the limited solubility of TMAT in UTREZ and MRPX at low temperatures have precluded its use. Studies have been conducted to find a solubilizing agent for TMAT which is acceptable for use in the propellant. At room temperature nitroparaffins and relatively nonpolar organic compounds do not dissolve TMAT. A variety of polar organic materials (alcohols, ketones, etc.) are effective solvents, but these are incompatible with NP. Chloroform and several other halogenated hydrocarbons of low molecular weight appear promising, but these compounds can be used only in low concentrations if ballistic performance is to be maintained. Furthermore, these solvents are quite volatile and would be expected to escape from the propellant even at reduced temperatures.

(U) Use of 15%w chloroform in UTREZ is required to dissolve TMAT. In accelerated testing (60° C) the gel time was found to be 15.6 hr compared to 7.2 hr for NTEB at an aziridine/carboxyl ratio of 1.3:1.0.

(U) Limited study has been conducted to find means of catalyzing the crosslinking reaction of carboxyl groups with aziridines. Use of certain metal salts and chelates appears promising.

(U) Preliminary studies showed that both zirconium acetyl-acetonate and chromium 2-ethylhexanoate catalyze the reaction of carboxyl groups with certain aziridines. The latter catalyst is particularly effective with UTREZ (table III).

(C) Such catalysis will be tested as a means of reducing the temperature required for curing the propellant. If the catalysts do not impair NP compatibility, an increase in propellant life can thereby be achieved.

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TABLE III
(U) CATALYSIS OF THE UTREZ/AZIRIDINE CURE REACTION^(a)

Aziridine Curative	COOH/Imine Equivalent Ratio	Chromium		Gel Time 60° C, hr
		2-Ethylhexanoate %w		
NTEB	1.1	1		3.5
NTEB	1.1	0		4.8
NTPB ^(b)	1.1	1		15.8
NTPB	1.1	0		23.3
TBM ^(c)	1.1	1		21.5
TBM	1.1	0		40.1

(a) All gumstock formulations contain 15%w MRPX plasticizer

(b) Nitrilotriethyl- β -propyleniminobutyrate

(c) Tris-(butylenimino)melamine

2. SOLID INGREDIENTS

a. Aluminum Fuel

(C) In the previous program, aluminum contents of propellants did not exceed 3.5%w. Because the maximum theoretical specific impulse of the NP/UTREZ/Al system requires an aluminum content of 15%w, it is desirable to conduct formulation and ballistic studies in this region. No adverse effects on propellant properties have been observed as the result of using 35 μ spheroidal aluminum in concentrations up to 16%w. Furthermore, no differences in propellant stability are observed when the aluminum is untreated, oven dried, or vacuum dried.

b. Nitronium Perchlorate

(C) It was originally intended to perform most of the propellant development work in this program using Reta-coated NP. However, as this material will be unavailable for an indefinite period emphasis has been shifted, at least temporarily, toward use of either uncoated or UTECOATED NP.

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(C) Studies conducted with Reta-coated oxidizer remaining from the previous program have shown conclusively that the coating deteriorates even when stored in an atmosphere containing 5 ppm or less of moisture and at a temperature of 28° C. A sample of 30-40 mesh NP with 4.4%w Reta coating (7402-66-5), which had been stored under the indicated conditions for ca 15 months, gassed within 20 hr of first contact with a standard binder formulation. Another sample of coated oxidizer (7402-123-5A) differing only in age (9 months) gassed in 9 days. Still another sample (402-123-5) of 9 months' age which had been stored at a fluctuating, but a lower average temperature gassed after 1 month. Microscopic examination of the poorest sample of Reta-coated NP revealed obvious deterioration and flaking of the coating. Interestingly, differential thermal analysis (DTA) of this sample of oxidizer showed an ignition temperature of 113° C compared with 96° C for the best sample. A possible explanation of this behavior is that extensive nitration of the deteriorated coating rendered it less reactive with NP.

(C) One year after their coating, the better samples were tested for hydrolysis after water immersion. Results are shown in table IV. Effect of coating deterioration on propellant properties is discussed more fully in section 4.0.

TABLE IV
(C) HYDROLYSIS OF RETA-COATED NP^(a)

<u>Hydrolysis</u>	<u>Sample 7405-123-5</u>	<u>Sample 7405-123-5A</u>
Original value, (b) 7 hr immersion in H ₂ O	<2%	<2%
Aged 1 year		
1-1/2-hr immersion	39%	87%
3-hr immersion	52%	93%

(a) 30-40 mesh, 4.4%w coating

(b) Determined by Union Carbide immediately after coating.

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(C) Batches of UTECOATED 30-40 mesh NP were prepared and evaluated in propellant formulations. Typical coating levels were 0.8%w (gravimetric determination) or 1.0 - 1.4%w (hydrolysis followed by acidimetric titration). Behavior of UTECOATED NP under DTA differed little from that of uncoated NP. No significant effects were observed at temperatures below 135° C. At this temperature an endotherm began and peaked at ca 145° C. A major exotherm began at 163° C and peaked at ca 175° C. In contrast, Reta-coated NP (4.4%w) exhibits an exotherm beginning in the range 75° to 90° C and culminating in ignition at 95° to 115° C. Undoubtedly, a major factor in the difference between UTECOATED and Reta-coated oxidizer is the greater fuel content of the heavier Reta coating. Properties of propellant formulations containing UTECOATED NP are discussed in section 4.0.

(C) The smallest particle size of NP which can be Reta-coated in a practical manner is in the range of 30-40 mesh. UTECOATING may be applied to smaller particles (perhaps as small as 100 mesh). However, the process becomes more difficult, and the weight of coating for a given level of protection increases directly with surface area of the oxidizer. In order to achieve optimum ballistic performance it would be most desirable to increase solids loading by incorporation of finely divided solid ingredients. A logical approach is to use as much of the relatively coarse NP as possible and then employ finely divided ammonium perchlorate (AP) and aluminum to provide a polymodal particle size distribution. On the basis of studies discussed in section 4.0, it appears that AP does not impair the stability of NP propellants.

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SECTION III FORMULATION STUDIES

(U) A variety of propellant formulations have been prepared to establish means of increasing ballistic performance (by way of increased solids loadings) and to provide specimens for stability and combustion studies. For the reader's convenience all formulations are identified in appendix A, which appears on a foldout sheet at the end of this report. All of the formulations were handmixed at a temperature of 28° C in an atmosphere containing less than 5 ppm of moisture.

(C) Initial studies of the effects of incorporating finely divided AP and increased concentrations of atomized aluminum in the basic propellant formulation (with Reta-coated NP) showed improved processability. Specifically, formulations UTX-8056, -8057, and -8058 mixed much more readily than did UTX-8054. After stability studies (reported in section 4.0) revealed no adverse effects resulting from use of AP and increased aluminum, formulations containing 75%w and 80%w solids were prepared. Formulation UTX-8164(75%w solids, 41.3%w NF, 17.7%w AP, 16%w Al) was observed to be more processable than UTX-8163(75%w solids, 59%w NP, 16%w Al), and UTX-8165(80%w solids, 44.8%w NP, 19.2%w AP, 16%w Al) was almost as easily handled as UTX-8163. Work to further increase solids loading was suspended when it was learned that Reta-coated oxidizer would not be available for an indefinite period. Emphasis was then placed on preparation of formulations to be used in studying the stability of propellants containing UTECOATED NP. Although no specific experiments were conducted to maximize solids loadings using UTECOATED NP, this form of the oxidizer appears to be about equivalent to Reta-coated material in terms of processability.

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SECTION IV

PROPELLANT STABILITY AND COMBUSTION

(C) In the preceding program it was established that machine-mixed propellants containing 56%w of Reta-coated (3.3%w) NP in UTREZ/NTEB/MRPX binder gave no evidence of gassing for 5 to 6 weeks when maintained at ca 30° C in an atmosphere containing less than 5 ppm moisture. When NP with 4.4%w Reta coating was used, gas was not observed until 8 weeks of storage. The most stable composition prepared was a hand-mixed, binder-rich sample containing NP with 4.4%w Reta coating. This sample was stored at 28° C in a dry box for 6 months before visible gassing occurred. In all of the foregoing cases the ages of the Reta coatings at the time of formulation were less than 3 months.

(C) All the Reta-coated oxidizer used in the present program was that remaining from the previous program, and significant deterioration of the coatings was observed. Formulation UTX-8055, which contained 1-year-old oxidizer but was otherwise identical to UTX-8054 (9-month-old oxidizer), proved to be quite unstable. UTX-8055 gassed within 24 hr of formulation and under DTA exhibited an exotherm beginning at 59° C and autoignition at 77° C. By comparison, UTX-8054 gassed in 9 days and under DTA exhibited an exotherm beginning at 66° C, and autoignition at 84° C. When UTX-8054 was reformulated with the same lot of NP after vacuum drying, the time to gas formation was increased to about 14 days. An equivalent formulation (UTX-8141) prepared with the best remaining sample of Reta-coated NP gassed after 1 month. Because appreciable deterioration of the Reta coatings continued during the present investigation, it was necessary to prepare control batches of propellant each time a study of ingredient variables was undertaken. The lack of a stable, standard formulation limits the generality of conclusions drawn from these studies.

(C) The effects of AP and increased aluminum content were studied in several series of formulations. Table V shows the results obtained with propellants containing Reta-coated NP of Lot 7405-123-5A. The slight increases in thermal stability observed in the formulations containing AP and increased aluminum may merely reflect reduced NP content. However, it can be concluded that AP and increased aluminum content do not impair stability. This conclusion is supported by the results of aging studies such as those shown in table VI.

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TABLE V
(U) EFFECT OF AMMONIUM PERCHLORATE
AND INCREASED ALUMINUM
ON INITIAL PROPELLANT STABILITY

	Formulation			
	UTX-8054	UTX-8056	UTX-8057	UTX-8058
Binder, %w	35.0	35.0	35.0	35.0
NP, Reta-coated, %w	63.0	44.1	49.0	34.3
Al, 35μ, %w	2.0	2.0	16.0	16.0
AP, to μ, %w	---	18.9	---	14.7
DTA, 5° C/min				
Exotherm inception, ° C	66	69	71	65
Autoignition, ° C	84	87	87	88

(C) On the basis of rate of visible reaction with uncoated NP, it appeared that squalane - a branched, paraffinic hydrocarbon - might be a suitable replacement for MRPX plasticizer. Propellant formulations were prepared using UTECOATED NP to compare the two plasticizers. The formulations and results of stability studies are shown in table VII. The superiority of MRPX over squalane is evident. Other conclusions drawn from these studies are (1) incorporation of AP (or perhaps the decreased concentration of NP) improves stability slightly; (2) propellants containing UTECOATED NP deteriorate much more rapidly than those containing even degraded Reta-coated NP; and (3) after sufficient deterioration, propellants containing UTECOATED NP burn explosively.

(C) The data shown in table VIII resulted from a study with two objectives (1) to compare two forms of UTECOATED NP with the best available (but nevertheless degraded) Reta-coated NP, and (2) to establish whether differential scanning calorimetry (DSC) provides any improvement over DTA in definition of propellant stability. Both propellants containing UTECOATED NP showed favorable initial properties, but their thermal stability decreased rapidly and reached a minimum 12 to 13 days after mixing. Thereafter, in spite of continuing visible deterioration, thermal stability returned to initial levels. Earlier studies have shown that freshly prepared propellants containing uncoated or UTECOATED NP burn stably unconfined or in micro-motors. However, after aging for about 1 week at 30° C in a dry atmosphere,

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TABLE VI
(U) EFFECT OF AMMONIUM PERCHLORATE AND INCREASED ALUMINUM
ON AGING STABILITY OF PROPELLANTS

Composition	Blinder, %w	NP, RETA-coated, %w	age of NP, mo.	AP, %w	Al, %w	Formulation						
						UTX-8141	UTX-8142	UTX-8143	UTX-8148	UTX-8149	UTX-8160	
Impact Sensitivity, kg cm	22	23	25	22	21	24	21	24	25	26	21	---
1 wk	24	25	21	24	25	24	25	25	26	27	21	---
5 wk												---
DTA, $\delta \cdot ^\circ C/min$												---
Exotherm inception, $^\circ C$	---	---	---	---	---	64	64	65	65	67	69	65
2 wk	64	62	63	64	62	63	64	63	63	62	62	61
3 wk	64	60	63	61	59	60	61	60	60	63	61	60
6 wk	59	60	63	61	61	60	60	60	60	60	60	60
9 wk	60	58	61	56	57	51	59	51	51	52	50	50
12 wk	55	56	57	52	60	ca55	55	55	55	55	55	55
14-1/2 wk												---
18 wk												---
Autobolition, $^\circ C$												---
2 wk	88	90	92	87	87	87	94	93	95	94	95	84
3 wk	86	88	92	83	85	83	85	89	89	90	89	85
6 wk	80	86	88	83	83	81	81	83	83	83	83	80
9 wk	82	84	93	78	82	82	83	86	86	88	88	82
12 wk	78	81	84	79	84	84	84	86	86	89	89	82
14-1/2 wk	80	75	85	75	75	75	75	75	75	75	75	75
18 wk												---
Characteristics at ca 16 wk												---
Gassing	Yes	No	No	Yes	Yes	Yes	Yes	Yes	No	No	No	---
Binders reversion	Medium	Slight	Very slight	Severe	Severe	Medium	Medium	Medium	Very slight	Very slight	Very slight	---
Unconfined combustion	Stable	Stable	Stable	Stable	Stable	Stable	Stable	Stable	Stable	Stable	Stable	---

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TABLE VII
 (U) EFFECT OF SQUALANE PLASTICIZER ON PROPELLANT STABILITY

Composition	Formulation			
	<u>UTX-8166</u>	<u>UTX-8167</u>	<u>UTX-8168</u>	<u>UTX-8169</u>
Plasticizer, 15%w of binder	MRPX	Squalane	MRPX	Squalane
Total binder, %w	25	25	25	25
NP, UTECOATED, %w	59	59	41.3	41.3
AP, %w	0	0	17.7	17.7
Al, %w	16	16	16	16
Impact Sensitivity, 4 days				
kg cm	27	28	>36	33
DTA, 5° C/min				
Exotherm inception, °C				
3 days	72	64	73	72
7 days	65	64	65	64
10 days	59	54	60	58
11 days	60	46	61	48
Autoignition, °C				
3 days	90	79	90	86
7 days	87	82	82	83
10 days	80	77	81	80
11 days	78	74	78	78
Combustion, unconfined, 11 days	Stable	Explosive	Stable	Explosive

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TABLE VIII
(C) STABILITY OF PROPELLANTS CONTAINING RETA-COATED AND UTECOATED NP

Composition	Formulation		
	UTX-8170	UTX-8171	UTX-8172
Binder, %w	25	25	25
Al, %w	16	16	16
NP, 30-40 mech			
UTECOAT-M, ca 0.79w			
RETA, 4.4%w			
UTECOAT-N, ca 0.79w			
DTA, 5 ° C/min, ° C			
Exotherm	Autoignition	Exotherm	Autoignition
4 days	74	86	53
6 days	68	83	53
10 days	55	76	62
12 days	49	72	63
13 days	49	75	62
19 days	64	84	66
25 days	76	90	74
46 days	75	89	74
DSC, ° C			
3 days	66	None	54
6 days	59	None	51
10 days	46	None	52
13 days	47	None	---
Combustion, unconfined			
25 days	Explosive	Stable	Explosive
46 days	Explosive	Stable	Explosive

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these propellants — even when unconfined — explode shortly after ignition. Explosive unconfined burning is not observed until the DTA exotherm inception temperature drops to about 55° C. The present studies indicate that recovery of thermal stability on aging is not accompanied by restoration of stable burning characteristics. For a time it was speculated that a transient chemical species might be responsible for explosive combustion. Now it appears that the responsible factors persist. Separation of the oxidizer from the binder by gas formation at the interface remains as one of the more likely causes.

(C) It appears significant that explosive unconfined combustion has never been observed with any propellant containing Reta-coated oxidizer, regardless of the state of degradation. It is also of interest that the thermal stability of propellant UTX-8171 increased steadily with age. In light of the degraded state of the Reta-coated NP in this propellant and the poor initial thermal stability, it is tempting to speculate that the thermal stability of propellants containing Reta-coated NP may also improve after passing through a minimum.

(D) Because DSC indicates lower exotherm temperatures than DTA, the former appears to be a more sensitive method for exploring propellant stability. Also, DSC offers the benefit of ease of manipulation, better control of sample environment, and reduced sample size. The probabilities that ignition does not occur during DSC are limited sample size and improved heat transfer.

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SECTION V

MECHANISM OF PROPELLANT DEGRADATION

(C) Samples of purified UTREZ prepolymer and MRPX plasticizer, as well as NP with degraded Reta coating, have been transmitted to Midwest Research Institute for use in study of the chemical reactions of NP, under Contract AF 04(611)-11199. This program includes the use of spectroscopic techniques to determine the rates of reaction and to characterize products resulting from contact of NP with various substrates, including model chemical compounds as well as UTC's binder ingredients. The results obtained under this program are completely consistent with UTC's ingredient and propellant stability studies. The following specific findings are especially pertinent to the present discussion:

- a. By ordinary (visual) standards, MRPX is among the most NP-compatible liquid hydrocarbons. Nevertheless, degradation products are spectroscopically detectable after 2 days of contact with NP at room temperature.
- b. A mixture of UTREZ and MRPX is even more reactive with NP.
- c. NTEB has a profound stabilizing effect on the propellant system. It is tentatively concluded that the basic NTEB molecule is adsorbed on the surface of NP and thereby prevents contact of the less polar MRPX and UTREZ with active sites on the NP.
- d. Treatment of NP with boron trifluoride (BF_3) does not reduce reactivity with the binder, while controlled treatment with water vapor greatly increases reactivity.
- e. Products of reaction of NP with the binder ingredients are initially confined to the interface.
- f. The rate of the degradation process is quite temperature sensitive.

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(C) These findings lend support to approaches being taken in the present program and suggest additional routes to improvement of propellant stability. One new approach is to employ NP in the purification of the binder and plasticizer. After a period of contact during which particularly reactive species interact with NP, the NP and adsorbed products may be removed physically. Another promising approach is to seek basic materials more effective than NTEB or cyclohexylpiperidine for the passivation of NP. Finally, it is desirable to establish the practical benefit of processing and storing the propellant at reduced temperatures.

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SECTION VI

FACILITIES

(U) During the previous program, ingredients were prepared and purified in ordinary laboratory equipment. Therefore, batch sizes were limited to the fractional-pound level. To provide sufficient supplies of ingredients for the present program, scaled-up equipment has been constructed. In addition, a surveillance facility has been constructed to permit storage of propellant at various temperatures in a dry atmosphere.

1. OXIDIZER TREATMENT

(C) Figure 1 shows the equipment employed to dry NP and other solid ingredients under vacuum. The process for drying NP is as follows.

(C) The oxidizer is transferred in a dry box to the heavy-walled Erlenmeyer flask visible at the lower left of figure 1. A closure consisting of an industrial joint, a stopcock and a tapered glass joint is attached to the flask and sealed by a Teflon O-ring and clamp. The closed assembly is attached to the vacuum line and the entire system is evacuated by a mechanical pump until the pressure is reduced to 2×10^{-2} torr. At this pressure the oil diffusion pump is started and the pressure is further reduced to ca 10^{-5} torr. Three traps cooled with liquid nitrogen prevent volatile materials from entering the pumps. Customarily, 100-gm lots of NP are dried for at least 16 hr at ambient temperature. At the end of the drying period the stopcock on the Erlenmeyer flask closure is closed so that no gas is admitted to the flask when the rest of the system is returned to atmospheric pressure. The still-evacuated flask is returned to the dry box before being opened.

2. UTREZ AND PLASTICIZER TREATMENT

(U) Details of the methods of purifying UTREZ and MRPX plasticizer were given in the final report covering the previous program.⁽¹⁾ The sequence of treatments for UTREZ are hypobromite oxidation, nitric acid oxidation, extraction with silica gel and vacuum stripping. For MRPX the sequence involves extraction with concentrated sulfuric acid, nitric acid oxidation, extraction with silica gel, vacuum distillation, and final drying over phosphorus pentoxide.

(1) Final Technical Progress Report, Contract No. AF 04(611)-9894,
Appendix A, p. 2-14.

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Figure 1. (C) Equipment for Drying NP

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(U) Figure 2 shows the reaction vessel and high-speed stirrer used for hypobromite oxidation of UTREZ. Also shown is a Rotavapor rotary vacuum evaporator which has been modified to permit use of dry-ice traps during solvent removal. This equipment, as well as that employed for the other indicated treatments, has been modified as necessary to ensure that the only materials in contact with the product being purified are glass, stainless steel, and Teflon. Where lubricants are required, Kel-F grease is used sparingly.

3. SURVEILLANCE

(U) Figure 3 is an interior view of a dry box which is equipped with a recirculating system capable of maintaining the moisture content below 5 ppm. The three cylindrical units are chambers in which propellant samples are stored at reduced temperatures. Each chamber consists of a metal cylinder of ca 15-in. ID which is wrapped with coils of copper tubing and enclosed in an insulating coating of polyurethane foam. The top closures are made of polystyrene foam. Refrigerant is provided for the three units by a single compressor and is admitted to the copper cooling coils through individual solenoid valves. Each solenoid valve is actuated by a thermistor-controlled relay. At present, the temperatures being maintained are 60°, 35°, and 10° F.

(U) Adequate workspace is available in the dry box so that propellants may be mixed and placed in storage without the possibility of exposure to atmospheric moisture.

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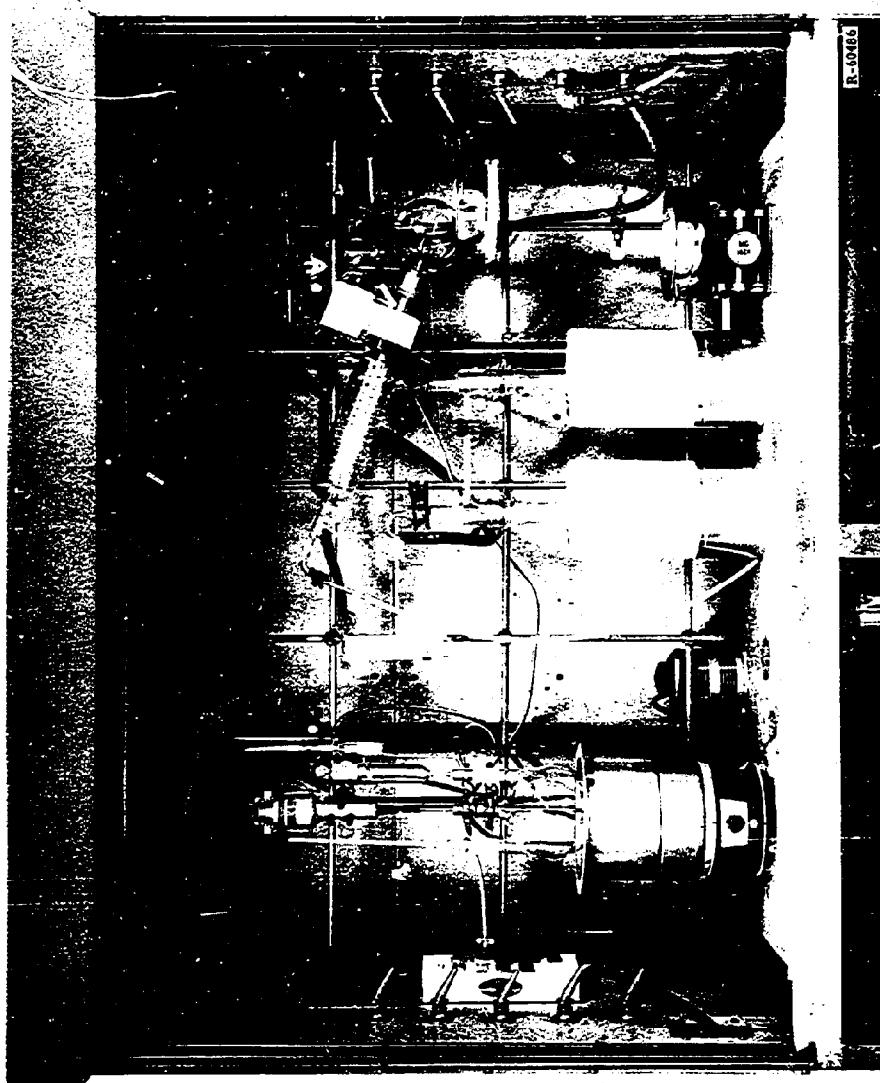


Figure 2. (U) Equipment Used in the Purification of UTREZ

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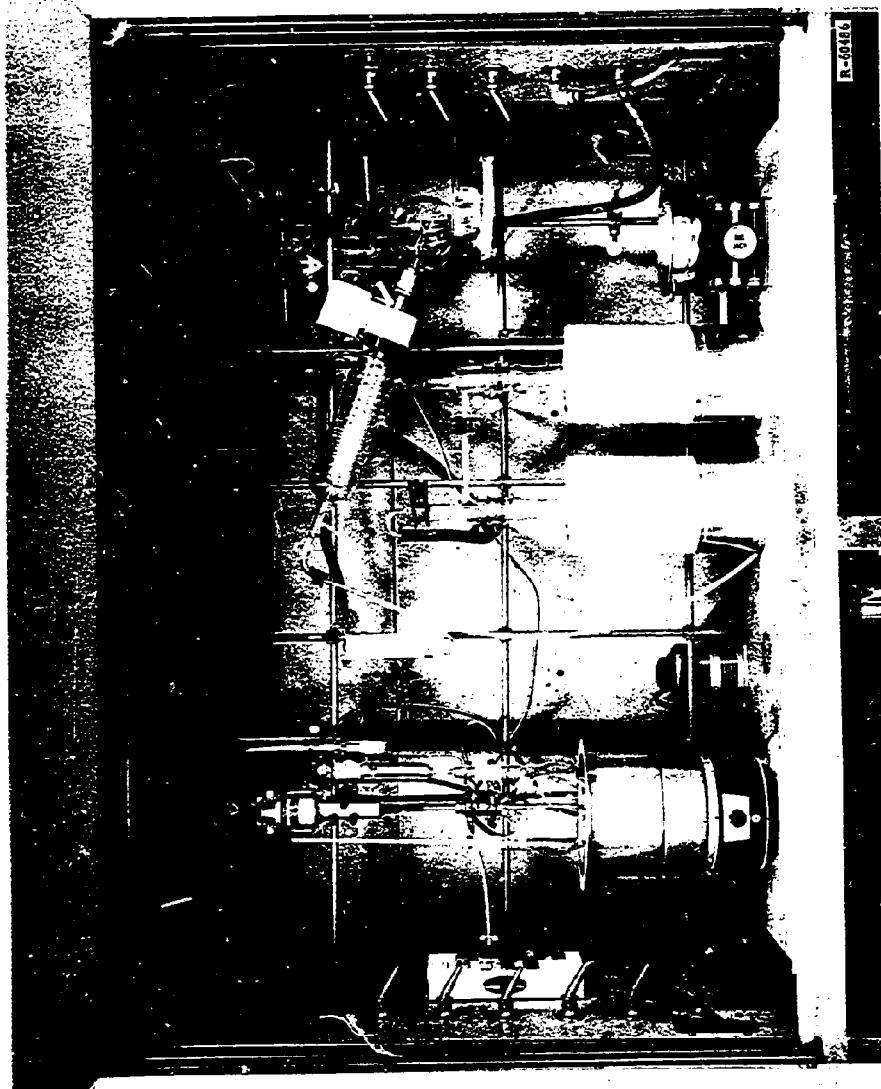


Figure 2. (U) Equipment Used in the Purification of UTREZ

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Figure 3. (U) Surveillance Facility

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SECTION VII

FUTURE ACTIVITY

(C) Pending the availability of Reta-coated NP, major emphasis will be placed on improvement and characterization of the stability of propellants containing uncoated and UTECOATED NP.

(U) Studies will include improved techniques for purification of ingredients, evaluation of alternate binder ingredients, and use of amine passivating agents. In addition, the feasibility of formulating and curing propellants at subambient temperatures will be explored. This, in turn, will require study of means of reducing binder viscosity and increasing cure reactivity.

(U) The most promising systems resulting from these studies will be tested for thermal stability and safety prior to testing in micromotors. If satisfactory properties are achieved, machine mixing, motor preparation, and test firings will be conducted at the 1-lb level.

(C) When Reta-coated, 30-40 mesh NP again becomes available, it will be employed at once in formulation optimization studies and motor testing at the 1-lb level.

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SECTION VIII

SUMMARY

(C) During this report period, activity was maintained at a low level pending technical redirection of the contract. Further curtailment of effort resulted from the unavailability of Reta-coated NP following an explosion in Union Carbide's coating facility.

(C) Pending availability of Reta-coated NP, emphasis has been placed on (1) improvement and characterization of the stability of propellants containing uncoated or UTECOATED NP, and (2) improvement of the ballistic performance of NP propellants by increasing solids loadings. By processing and storing the propellants at reduced temperatures significant improvement in stability is expected. However, this reduces the rate of cure of the binder and increases its viscosity. The latter effect increases the difficulty of increasing solids loading. To counter these undesirable effects, improved plasticization of the binder and cure catalysis have been attempted. A low viscosity isoparaffin, 2,6,10,14-tetramethylpentadecane, and several nitroparaffins have shown promise as improved plasticizers. Cure catalysis has been effected by use of chromium 2-ethylhexanoate. Increased solids loadings have been achieved without impairment of stability by use of increased concentrations of aluminum and by incorporation of finely divided AP as a supplementary oxidizer.

(C) Hydrolysis tests and propellant stability studies have revealed that Reta-coated NP deteriorates markedly in 1 year under mild storage conditions (ambient temperature, less than 5 ppm atmospheric moisture). The useful life of propellants containing UTECOATED NP appears to be not more than 1 week under these conditions. Such propellants reach a minimum thermal stability in 10 to 14 days and then regain stability at an age of about 4 weeks. These propellants exhibit explosive combustion after about 1 week and do not regain stable burning characteristics even when DTA indicates recovery of thermal stability.

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(C) Equipment has been constructed for scaled-up drying of NP and for purification of UTREZ prepolymer. Approximately 5 lb of prepolymer has been purified and is available for formulation studies. A surveillance facility has been constructed to permit study of propellant stability at three reduced temperatures in a dry environment.

(C) Future activity will emphasize means of improving propellant stability by more rigorous purification of ingredients, oxidizer passivation, and use of reduced temperatures for processing and storage.

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APPENDIX A
PROPELLANT FORMULATIONS

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	UTX-8054	UTX-8055	UTX-8056	UTX-8057	UTX-8058	UTX-8
Ingredients						
UTREZ, 1.0 eq (all)	---	---	---	---	---	---
NTEB, 1.2 eq (all)	---	---	---	---	---	---
MRPX, %w	5.25	5.25	5.25	5.25	5.25	5.2
Squalane, %w	---	---	---	---	---	---
Total Binder, %w	35.00	35.00	35.00	35.00	35.00	35.0
NP, 30-40 mesh, %w						
Uncoated	---	---	---	---	---	---
Reta, 4.4%w (7402-66-5)	---	63.00	---	---	---	---
Reta, 4.4%w (7405-123-5)	---	---	---	---	---	63.0
Reta, 4.4%w (7405-123-5A)	63.00	---	44.10	49.00	34.30	---
UTECOAT-M, ca 0.7%w	---	---	---	---	---	---
UTECOAT-N, ca 0.7%w	---	---	---	---	---	---
AP, ca 10μ, %w	---	---	18.90	---	14.70	---
Al, ca 35μ, %w	2.00	2.00	2.00	16.00	16.00	2.0
Total Solids, %w	65.00	65.00	65.00	65.00	65.00	65.

	UTX-8162	UTX-8163	UTX-8164	UTX-8165	UTX-8166
Ingredients					
UTREZ, 1.0 eq (all)	---	---	---	---	---
NTEB, 1.2 eq (all)	---	---	---	---	---
MRPX, %w	5.25	3.75	3.75	3.00	3.75
Squalane, %w	---	---	---	---	---
Total Binder, %w	35.00	25.00	25.00	20.00	25.00
NP, 30-40 mesh, %w					
Uncoated	---	---	---	---	---
Reta, 4.4%w (7402-66-5)	---	---	---	---	---
Reta, 4.4%w (7405-123-5)	31.50	59.00	41.30	44.80	---
Reta, 4.4%w (7405-123-5A)	---	---	---	---	59.00
UTECOAT-M, ca 0.7%w	---	---	---	---	---
UTECOAT-N, ca 0.7%w	---	---	---	---	---
AP, ca 10μ, %w	31.50	---	17.70	19.20	---
Al, ca 35μ, %w	2.00	16.00	16.00	16.00	16.00
Total Solids, %w	65.00	75.00	75.00	80.00	75.00

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Formulation No.								
	UTX-8058	UTX-8141	UTX-8142	UTX-8143	UTX-8148	UTX-8149	UTX-8160	UTX-8161
	---	---	---	---	---	---	---	---
	---	---	---	---	---	---	---	---
5.25	5.25	5.25	5.25	5.25	5.25	5.25	5.25	5.25
---	---	---	---	---	---	---	---	---
35.00	35.00	35.00	35.00	35.00	35.00	35.00	35.00	35.00
	---	---	---	---	---	---	---	---
	---	---	---	---	---	---	---	---
	---	63.00	49.00	34.30	63.00	49.00	44.00	34.30
34.30	---	---	---	---	---	---	---	---
---	---	---	---	---	---	---	---	---
---	---	---	---	---	---	---	---	---
14.70	---	---	14.70	---	---	---	19.00	14.70
16.00	2.0	16.00	16.00	2.00	16.00	2.00	16.00	
65.00	65.00	65.00	65.00	65.00	65.00	65.00	65.00	65.00

Formulation No.								
-8165	UTX-8166	UTX-8167	UTX-8168	UTX-8169	UTX-8170	UTX-8171	UTX-8172	
	---	---	---	---	---	---	---	---
	---	---	---	---	---	---	---	---
.00	3.75	---	3.75	---	3.75	3.75	3.75	3.75
---	---	3.75	---	3.75	---	---	---	---
.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00
	---	---	---	---	---	---	---	---
	---	---	---	---	---	---	---	---
.80	---	---	---	---	---	59.00	---	---
---	---	---	---	---	---	---	---	---
.	59.00	59.00	41.30	41.30	59.00	---	---	59.00
---	---	---	---	---	---	---	---	---
.20	---	---	17.70	17.70	---	---	---	---
.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00	16.00
.00	75.00	75.00	75.00	75.00	75.00	75.00	75.00	75.00

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Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

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		2b. GROUP 4
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4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Technical Report AFRPL-TR-66-154, 1 June 1965 through 31 May 1966		
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11. SUPPLEMENTARY NOTES N/A	12. SPONSORING MILITARY ACTIVITY Air Force Rocket Propulsion Laboratory Research and Technology Division Air Force Systems Command, USAF Edwards, California	
13. ABSTRACT (Unclassified)		
<p>A 24-month program is being conducted to produce practical, castable, composite solid propellants containing a high-energy oxidizer. Emphasis is being placed on improving the thermal stability and increasing the attainable solids loadings of propellants containing particulate oxidizer, a compatible binder (carboxy-terminated polyisobutylene), and polyfunctional aziridiny crosslinking agents. (U)</p> <p>Work during this report period was directed toward improving propellant processability at reduced temperatures. Promising plasticizers, including hydrocarbons and nitroparaffins, were evaluated. Catalysis of the carboxy-aziridine crosslinking reaction was demonstrated. Equipment was constructed for purification of ingredients and for stability tests at reduced temperatures. Supplies of purified ingredients were prepared. (U)</p>		

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Solid propellants High-energy solid oxidizer Carboxy-terminated polyisobutylene						
INSTRUCTIONS						
1. ORIGINATING ACTIVITY: Enter the name and address of the contractor, subcontractor, grantees, Department of Defense activity or other organization (corporate author) issuing the report.	imposed by security classification, using standard statements such as:					
2a. REPORT SECURITY CLASSIFICATION: Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.	(1) "Qualified requesters may obtain copies of this report from DDC."					
2b. GROUP: Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.	(2) "Foreign announcement and dissemination of this report by DDC is not authorized."					
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6. REPORT DATE: Enter the date of the report as day, month, year, or month, year. If more than one date appears on the report, use date of publication.	If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.					
7a. TOTAL NUMBER OF PAGES: The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.	11. SUPPLEMENTARY NOTES: Use for additional explanatory notes.					
7b. NUMBER OF REFERENCES: Enter the total number of references cited in the report.	12. SPONSORING MILITARY ACTIVITY: Enter the name of the departmental project office or laboratory sponsoring (paying for) the research and development. Include address.					
8a. CONTRACT OR GRANT NUMBER: If appropriate, enter the applicable number of the contract or grant under which the report was written.	13. ABSTRACT: Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.					
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